Specifications of Fatty Acid Composition for Identification of Fats and Oils by Gas Liquid Chromatography

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The Codex Committee on Fats and Oils of the Joint Food and Agricultural Organization of the United Nations and the World Health Organization (FAO/WHO), Codex Alimentarius Commission, has for several years had under consideration a complete revision of their standards for the identification of fats and oils. As early as 1966, the Canadian Delegation proposed that the classical method be replaced by an alternate system based on the fatty acid composition of each fat or oil as determined from gas liquid chromatographic patterns. The classical method is based on the identification of a specific fat or oil by means of a combination of its iodine value, relative density, refractive index and saponification value.

Prior to the third meeting of the Fats and Oil Committee held in London April 24–28, 1967, the Canadian Delegation submitted a proposal for the replacement of the classical method with a gas liquid chromatographic (GLC) procedure. In this proposal the Canadian Delegation discussed disadvantages of the classical system, particularly the requirement for a heavy investment of analytical time and skill in many techniques, and inability, even when all four indices are considered, to resolve several admixtures containing common oils of commerce. At the same time, advantages of the gas liquid chromatographic procedure were emphasized, including the fact that it permits identification of oils which cannot be identified by the classical method and offers promise of resolving many additional pairs of oils in admixture. Furthermore, since it requires only one analysis, it can be made quite rapidly and applies equally well to refined and unrefined oils, necessitating only one set of standards.

From the beginning, at least as early as 1966, the United States supported the Canadian proposal in principle and recommended specifically that the Codex Committee on Fats and Oils urge the Codex Committee on Methods of Analysis to evaluate the current status of the science of gas liquid chromatography and, if feasible, to adopt GLC procedures that can be used by the Codex Committee on Fats and Oils.

At the fourth meeting of the Codex Committee on Fats and Oils, sponsored by the Joint FAO/WHO Codex Alimentarius Commission and held in London, April 24–28, 1967, the Canadian proposal was presented. The U.S. position on the proposal was one of partial support. This position was explained: "Full support of the Canadian proposal would not be justified, since most available evidence indicates that adoption of the proposal at this time would be premature and would lead to serious practical difficulties. Full opposition to the proposal would place the United States in a position of being ultra-conservative and unwilling to recognize the most modern scientific advances in the field of fat and oil analysis. The U.S. delegation therefore took the position that a system of identification based on GLC, in addition to the classical system, be carried in the draft standards at this stage. This would be done with the expectation that before final promulgation of the standards, the science of GLC will have progressed to a point where the present uncertainties concerning it would be eliminated and that it would provide a far more accurate means of identification than is possible with the present classical procedures. If this expectation should be realized, the classical system could then be dropped before final promulgation. The U.S. delegation argued that acceptance of its views would encourage the necessary international cooperation needed in the further perfection of the GLC system of oil and fat identification." After considerable discussion this U.S. position prevailed among the delegates from the 21 nations represented at this fourth meeting.

In comments on the Canadian proposal, submitted to delegates of the countries to be represented prior to the April 1967 Meeting, the United States delegation had presented its position, as given above. With their comments the delegation commented on the requirement for GLC standards and proposed to solicit recommendations concerning such standards from the Codex Committee on Methods of Analysis which would likely be based on the work of the International Union of Pure and Applied Chemistry. In these remarks they commented that the American Oil Chemists' Society, through its Instrumental Techniques Committee and the Gas Chromatography Subcommittee thereof, was currently very active in this field and "could probably render valuable assistance to the Codex Committee on Methods of Analysis" hopefully prior to the April 1967 meeting.

The hope for accomplishment of this objective prior to the April 1967 meeting was apparently not realized, but shortly after acceptance of its proposal at this meeting, the United States delegation, through its Chairman Lawrence Zeleny, contacted the Society with a request for assistance in the preparation of such standards. The AOCS Executive Director, C. H. Hauber, arranged a meeting which was held during the joint AACC-AOCS 59th Annual Con-

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TABLE I

Arachis Oil[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<0.5
Myristic	14:0	<1.0
Palmitic	16:0	6.0–14
Stearic	18:0	2.0– 6.5
Arachidic	20:0	1.0– 2.0
Behenic	22:0	2.0– 4.0
Lignoceric	24:0	1.0– 2.0
Palmitoleic	16:1	<1.0
Oleic	18:1	40 –72
Linoleic	18:2	13 –38
Linolenic	18:3	<0.50
Eicosenoic	20:1	0.5– 1.5

[a] Synonyms: peanut, groundnut.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

TABLE II

Cottonseed Oil[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<0.5
Myristic	14:0	0.5– 2.0
Palmitic	16:0	17 –29
Stearic	18:0	1.0– 4.0
Arachidic	20:0	<0.5
Behenic	22:0	....
Lignoceric	24:0	....
Palmitoleic	16:1	<1.5
Oleic	18:1	13 –44
Linoleic	18:2	33 –58
Linolenic	18:3	<0.5
Eicosenoic	20:1	<0.5

[a] Synonyms: none.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

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TABLE III
Maize Oil[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<0.5
Myristic	14:0	<1.0
Palmitic	16:0	8 –19
Stearic	18:0	0.5– 4.0
Arachidic	20:0	<1.0
Behenic	22:0	....
Lignoceric	24:0	....
Palmitoleic	16:1	<1.0
Oleic	18:1	19 –50
Linoleic	18:2	34 –62
Linolenic	18:3	<1.0
Eicosenoic	20:1	<0.5

[a] Synonyms: corn.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

TABLE IV
Mustardseed Oil[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<0.5
Myristic	14:0	<1.0
Palmitic	16:0	0.5– 4.5
Stearic	18:0	0.5– 3.0
Arachidic	20:0	<1.5
Behenic	22:0	0.2– 2.5
Lignoceric	24:0	<0.5
Palmitoleic	16:1	<1.0
Oleic	18:1	8.0–23.0
Linoleic	18:2	10.0–24.0
Linolenic	18:3	6.0–18.0
Eicosenoic	20:1	4.0–13.0
Erucic	22:1	22.0–49.0
Docosadienoic	22:2	<1.0
Tetracosenoic	24:1	0.5– 2.5

[a] Synonyms: none.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

TABLE V
Rapeseed Oil[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<0.5
Myristic	14:0	<1.0
Palmitic	16:0	0.5– 5.0
Stearic	18:0	0.5– 3.0
Arachidic	20:0	<1.5
Behenic	22:0	<1.5
Lignoceric	24:0	<2.0
Palmitoleic	16:1	<1.0
Oleic	18:1	9.0–25
Linoleic	18:2	11 –25
Linolenic	18:3	5.0–12
Eicosenoic	20:1	5.0–15
Erucic	22:1	30 –60
Docosadienoic	22:2	<1.0

[a] Synonyms: colza, ravison, sarson, toria, turnip rape.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

TABLE VI
Safflower Seed Oil[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<0.5
Myristic	14:0	<1.0
Palmitic	16:0	2.0–10
Stearic	18:0	1.0– 6.0
Arachidic	20:0	<1.0
Behenic	22:0	<1.0
Lignoceric	24:0	....
Palmitoleic	16:1	<0.5
Oleic	18:1	7.0–40
Linoleic	18:2	55 –80
Linolenic	18:3	<1.0
Eicosenoic	20:1	<0.5

[a] Synonyms: carthamus, durdee.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

vention in the Washington-Hilton Hotel, Washington, D.C., Tuesday, April 2, 1968. The minutes of this meeting show that it was attended by five United States or Canadian delegates or advisors to the delegates to the Codex Committee on Fats and Oils, by four members of the American Oil Chemists' Society, and by two members from American industrial organizations.

At this meeting the requirements of the Codex Committee were set forth by the U.S. Delegates. This delegation had felt in the past that the proposal to use GLC in Codex standards had been premature because of insufficient knowledge of the ranges in fatty acid composition characteristics of some of the fats and oils. However recent developments in this field had led them to the opinion that Codex standards should utilize GLC analysis at least on an optional basis, otherwise standards based entirely on the classical fat and oil characteristics would soon become obsolete.

It was emphasized by the AOCS members that the major problem in establishing such standards is the need for a comprehensive survey of the variability in fatty acid distribution for each product for which standards are to be developed in order to establish completely valid and at the same time reasonable minimum and maximum requirements for such standards. It was agreed that the Codex delegation would furnish a list of the fats and oils for which standards were now required and that the AOCS, through its Instrumental Techniques Committee, would initiate the necessary survey to establish the minimum and maximum limits for each fatty acid in each specific fat or oil.

It was also agreed that in establishing these standards, the range for each fatty acid in each fat or oil would be broad enough to include all products ordinarily encountered in world trade, the complete range expected for commercial fats or oils, but not necessarily broad enough to include all products which might be encountered under exceptional conditions. Exceptional oils, it was pointed out, can result from agronomic or genetic advances to impart special properties to a specific oil. Three examples are given to illustrate such exceptional oils (where the minimum or maximum concentration or both of one or more acids might be outside of the normal range for oils of commerce.)

1. Safflower oil can be of high linoleic or high oleic variety. In the former, the 18:2 acid would be 55% to 80% and 18:1 7.0% to 40%; in the latter, the 18:2 would be 7.0% to 40% and 18:1 55% to 80%. To cover both, 18:1 would be 7.0% to 80% and 18:2 also 7.0% to 80%.
2. A rapeseed oil has now been developed which contains no erucic acid (22:1) so if this unusual variety was included, this component would be 0% to 60%, 22:1 instead of 50% to 60%.
3. The fatty acid composition for arachis oil has been found to be "unusual" in the following cited examples: Arachidic was found to be less than 1% in eight of 11 varieties studied (1). Lignoceric was found to be less than 1% in one out of three samples investigated by Craig and Murthy; for one out of seven varieties (2), and one out of 22 samples (3).

The delegates to the Codex Committee on Fats and Oils requested specifications for the following fats and oils: arachis oil (peanut oil) (groundnut oil), cottonseed oil, maize oil (corn oil), rapeseed oil, safflower oil, sesame seed oil, soya bean oil (soybean oil), sunflower seed oil, lard (rendered pork fat), and premier jus (edible tallow). Mustard seed oil was added to this list after the fifth meeting of the Codex Committee on Fats and Oils in London, September 16–20, 1968.

The Chairman of the Gas Chromatography Subcommittee of the Instrumental Techniques Committee prepared a list, from an exhaustive search of the literature, of the minimum-maximum range of fatty acid composition, as obtained by gas chromatography. These values were submitted to several

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• *Fatty Acid Composition by TLC* . .

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TABLE VII
Sesame Seed Oil[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<0.5
Myristic	14:0	<0.5
Palmitic	16:0	7.0–12
Stearic	18:0	3.5– 6.0
Arachidic	20:0	<1.0
Behenic	22:0	<1.0
Lignoceric	24:0	....
Palmitoleic	16:1	<0.5
Oleic	18:1	35 –50
Linoleic	18:2	35 –50
Linolenic	18:3	<1.0
Eicosenoic	20:1	<0.5

[a] Synonym: gingelly.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

TABLE VIII
Soya Bean Oil[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<0.5
Myristic	14:0	<0.5
Palmitic	16:0	7.0–12
Stearic	18:0	2.0– 5.5
Arachidic	20:0	<1.0
Behenic	22:0	<0.5
Lignoceric	24:0	....
Palmitoleic	16:1	<0.5
Oleic	18:1	20 –50
Linoleic	18:2	35 –60
Linolenic	18:3	2.0–13
Eicosenoic	20:1	<1.0

[a] Synonym: soybean.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

TABLE IX
Sunflower Seed Oil[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<0.5
Myristic	14:0	<0.5
Palmitic	16:0	4.0– 9.0
Stearic	18:0	1.0– 6.0
Arachidic	20:0	<1.0
Behenic	22:0	<1.0
Lignoceric	24:0	....
Palmitoleic	16:1	<0.5
Oleic	18:1	14 –70
Linoleic	18:2	20 –75
Linolenic	18:3	<0.5
Eicosenoic	20:1	<0.5

[a] Synonym: none.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

TABLE X
Lard (and Rendered Pork Fat)[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<1.0
Myristic	14:0	0.5– 2.5
Palmitic	16:0	20 –32
Stearic	18:0	5.0–18
Arachidic	20:0	<1.0
Behenic	22:0	<0.5
Lignoceric	24:0	....
Palmitoleic	16:1	2.0– 4.0
Oleic	18:1	35 –60
Linoleic	18:2	3.0–15
Linolenic	18:3	<2.0
Eicosenoic	20:1	<1.0

[a] Synonym: none.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

TABLE XI
Premier Jus (and Edible Tallow)[a,b]

Acids	Carbon atoms	Range
Saturated	Less than 14	<1.0
Myristic	14:0	1.0– 8.0
Palmitic	16:0	23 –37
Stearic	18:0	6.0–20
Arachidic	20:0	<1.0
Behenic	22:0	....
Lignoceric	24:0	....
Palmitoleic	16:1	1.5– 6.0
Oleic	18:1	36 –50
Linoleic	18:2	0.5– 5.0
Linolenic	18:3	<1.0
Eicosenoic	20:1	<1.0

[a] Synonyms: none.
[b] Fatty acid composition by gas chromatography (wt % of methyl esters).

members of AOCS knowledgeable in fatty acid composition of fats and oils and from their remarks, comments and suggestions final ranges were selected as GLC specifications.

These specifications were submitted to the U.S. Delegation to the Codex Committee on Fats and Oils, through their Chairman, Dr. Zeleny, and presented at the fifth session of the Codex Committee on Fats and Oils, sponsored by the Joint FAO/WHO Codex Alimentarius Commission and held in London September 16–20, 1968. The specifications were, according to the minutes of this meeting, well received. However, as the delegates had not had an opportunity to study them, the United States proposal ". . . that all of the Codex standards for specified kinds of fats and oils contain two alternative sets of identification requirements, one based on the classical physical and chemical characteristics and the other on fatty acids composition as determined by GLC," while supported by a majority of the delegates, was referred by the Chairman of the Fifth Session back to the delegates with the request for ". . . all participating countries to study the U.S. proposal further and to consider at the next meeting of the Committee whether GLC procedures for identifying fats and oils should be inserted into the standards as 'advisory criteria.'"

At this Fifth Session of the Codex Committee on Fats and Oils, standards for mustard seed oil were discussed. One outcome of this discussion was a request to the AOCS for inclusion of standards for this oilseed, from GLC measurements. These were subsequently furnished to M. H. Neustadt, who succeeded Lawrence Zeleny as Chairman of the U.S. Delegation.

In the meantime considerable interest has been made evident by several members of AOCS, usually with requests for the GLC specifications. It appears that the most suitable medium to make the specifications available to any interested member of the Society, while they are being considered by the Codex Committee on Fats and Oils, would be by their publication in the *Journal*.

Complete specifications for the 11 oilseeds thus far considered, including mustard seed oil, are given in the attached Tables.

REFERENCES

1. French, R. V., JAOCS *39*, 176 (1962).
2. Worthington, R. E., and K. T. Holley, JAOCS *44*, 515 (1967).
3. Iverson, J. L., D. Firestone and W. Horwitz, J. Assoc. Offic. Anal. Chemists *46*, 718 (1963).

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Fatty Acids Statistics

Production of animal, vegetable, and marine fatty acids totalled 51.7 million pounds in January 1970. Inclusion of the tall oil types raised the overall January production level to 84.2 million pounds.

Disposition of fatty acids amounted to 64.7 million pounds in January. Including tall oil fatty acids, January 1970 disposition totalled 97.6 million pounds.

Stocks of fatty acids other than the tall oil types amounted to 39.7 million pounds on January 31st, down 7.7 million pounds from January 1st.

Source: Fatty Acid Producers' Council, 485 Madison Ave., New York, N.Y. 10022.